UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

APR 18 1984

OFFICE OF
SOLID WASTE AND EMERGENCY RESPONSE
RE: WIBFSJ0240

Mr. John Angiolini
Givaudan Corporation
125 Delawanna Avenue
Clifton, NJ 07014

Dear Mr. Angiolini,

Pursuant to our telephone conversation of April 9, I am enclosing the engineering analysis of the Hexachlorophene (HCP) manufacturing process performed for us by Radian Corporation. We would appreciate your review of this document. In particular, we would like you to verify its accuracy as it applies to your manufacturing process.

In addition, we request your comments on the following:

1. What are the specifications of the 2,4,5-TCP used for the manufacture of pharmaceutical and pesticide grade HCP?

2. Please provide us with analytical data presently available to you for concentrations of dibenzofurans, and, tetra-, penta-, and hexachlorodioxins, including 2,3,7,8-TCDD in a) 2,4,5-TCP feedstock; b) HCP products, and c) the waste streams. If no analytical data is available please exercise your best judgement in estimating the levels of concentration (to the ppb level).

3. Please tell us whether you re-crystallize after the first crystallization product, and whether the mother liquid is discarded or re-used, and, if the latter, how often it is recycled.

4. Did Radian Corporation correctly identify the process flow and waste streams? If not, please correct.

5. Is wastewater from the HCP process independently disposed, or is it co-mingled with wastewater from other processes? How is it disposed (e.g., settling pond, discharged (where?)) with or without treatment? If the latter, what treatment?

If you have any questions, please call our contractor, Dr. Shri Kulkarni at Radian Corporation (919-541-9100). Thank you in advance for your cooperation. We look forward to your early reply.

Sincerely,

Francine S. Jacoff
Program Manger
Listing Program

Enclosure

GIVAUDAN CORPORATION

125 Delawanna Avenue
Clifton, New Jersey 07014
Phone: (201) 546-8000
Cable: Givaudanco, Clifton
Telex: 138901

April 30, 1984

Dr. Shrikant V. Kulkarni
Radian Corporation
3200 E. Chapel Hill Road
Nelson Highway
Research Triangle Park, N. C. 27709

Dear Dr. Kulkarni:

I have just received a copy of the engineering analysis of the Hexachlorophene (HCP) manufacturing process prepared by the Radian Corporation for Dr. Judy Bellin of the United States Environmental Protection Agency. In reviewing your report, numerous factual errors have been found regarding the industrial process for the synthesis of Hexachlorophene from 2,4,5-Trichlorophenol. As such, I would urge you to advise Dr. Bellin to delay publishing or distributing the Final Draft until these errors are corrected.

Most significant of these errors is the assumption that the synthesis/purification of the pharmaceutical grade of Hexachlorophene invloves the preparation of the sodium salt of Hexachlorophene prior to recrystallization. Based on this assumption it is stated that, "...In the pharmaceutical grade process, conditions are more favorable for the formation of TCDD...". As the only current supplier of USP grade Hexachlorophene in the United States, Givaudan does not prepare the sodium salt (or any other alkaline salt) of Hexachlorophene as part of its purification procedure. Furthermore, Givaudan for many years has been producing Hexachlorophene which meets USP specifications without the need for a crystallization step as part of its purification process.

While a thorough review of the report will take some time, it would seem that such a delay would be worthwhile in the interest of accuracy. I will call you regarding the status of our review by May 15. If, prior to this, you have any questions, please do not hesitate to call me.

Very truly yours,
GIVAUDAN CORPORATION

H A Brandman

Dr. H. A. Brandman
(201) 365-8485

cc: Dr. Judy Bellin
Ms. Francine S. Jacoff
U.S.E.P.A.

GIVAUDAN CORPORATION

125 Delawanna Avenue
Clifton, New Jersey 07015-503
Phone: (201) 365-8485
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June 19, 1984

Dr. Judy Bellin
U.S. Environmental Protection Agency
Office of Solid Waste (WH-565B)
Washington, D.C. 20460

Dear Dr. Bellin:

Since my initial review of the Radian Corporation's Study, the Givaudan Corporation has ceased to be a manufacturer of Hexachlorophene (HCP). As such, I have not undertaken a detailed analysis of their report, "Waste Streams From Hexachlorophene Manufacturing Processes."

During the period of time that the Givaudan Corporation did produce Hexachlorophene, however, the basic process employed was essentially as described on p. 3-2 of Dr. Kulkarni's report; that is, the sulfuric acid catalyzed condensation of 2,4,5-trichlorophenol with paraformaldehyde in ethylene dichloride. However, as was stated in my original letter to Dr. Kulkarni, during the time that Givaudan produced HCP, the product which was obtained from this process met the standards of the U.S. Pharmacopeia without the need to either prepare an alkaline salt of the HCP or crystallize/recrystallize the finished product. Consequently, the statement (p. 2-2) that "...wastes from the manufacture of pharmaceutical grade hexachlorophene are expected to have a higher dioxin content because of the high purity requirement of the product..." is untrue based on the process for manufacturing HCP at Givaudan.

With regard to Ms. Jacoff's letter of April 18, 1984 to Mr. Angiolini, the following information applies to the questions in the same sequence as they were listed:

1.) Increasing concern regarding the toxicity of 2,3,7,8-TCDD (and improved analytical techniques) led in recent years to a gradual tightening of the specifications of the 2,4,5-trichlorophenol purchased by the Givaudan Corporation. In the period preceding the cessation of HCP manufacture by Givaudan, the TCDD specification was < 1 ppb. A copy of the Givaudan Quality Control Department's specifications for 2,4,5-trichlorophenol is attached.

GIVAUDAN CORPORATION

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2.) We do not have any analytical data available regarding the possible presence of chlorinated dibenzofurans or dioxins OTHER than 2,3,7,8-tetrachlorodibenzodioxin (2,3,7,8-TCDD). Analyses of HCP sold by Givaudan as well as the waste streams from the manufacturing process showed no detectable 2,3,7,8-TCDD as the limit of detection (1 ppb) for the analytical procedure employed (gas chromatography-mass spectrometry).

3.) As mentioned above, the HCP produced and sold by Givaudan was not purified by crystallization.

4.) See above.

5.) Wastewater from the HCP process was co-mingled with wastewater from other processes. All such wastewater was/is neutralized prior to treatment by the regional Passaic Valley Sewage Treatment Facility.

I hope that this information regarding HCP manufactured by the Givaudan Corporation is of some assistance to you. If I can be of further help, please do not hesitate to contact me.

Very truly yours,

Givaudan Corporation

H.A. Brandman

Dr. H.A. Brandman

:da

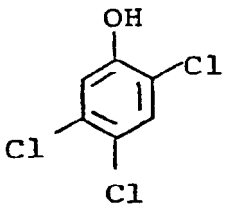
cc: Dr. S.V. Kulkarni
Ms. Francine S. Jacoff

GIVAUDAN CORPORATION

QUALITY CONTROL DEPARTMENT

SPECIFICATIONS

2,4,5-TRICHLOROPHENOL



Molecular Formula: $C_6H_3Cl_3O$

Molecular Weight: 197.46

APPEARANCE & COLOR: White to off-white fused mass or flake. Molten material may have a slight pink cast.

COLOR OF TCP IN SOLUTION: A 10% alcoholic solution is prepared. The color of this solution is no darker than Givaudan Color 3 (.00050 g. Potassium Dichromate/100 ml. 2% Sulfuric Acid).

CONGEALING POINT: 63.5°C. Minimum
Givaudan Index Test Method V.

PURITY & IMPURITES BY GAS CHROMATOGRAPHIC ANALYSIS:

2,4,5-Trichlorophenol	98.7% Minimum
2,3,6-Trichlorophenol	0.5% Maximum
Dichlorophenols	0.5% Maximum
2-Chloro-5-Methoxyphenol	0.3% Maximum
Dichloromethoxy Phenol	0.5% Maximum

Procedure: GLC analysis using normalized area for the determination.

Equipment:

1. GLC unit equipped with a flame ionization, detector and electronic integrator.
2. Column: 6' x 1/8", 4% FFAP on 100/120 chromosorb G.
3. Chromatographic Conditions
   - A. Flow: 40 ml/min.
   - B. Sample Size: 0.2 ul of a solution of sample in acetone.
   - C. Sensitivity: 2×10^2
   - D. Oven Temperature: 190°C.

Note: Identities and purities are determined based on the attached curve.

SPECIAL REQUIREMENTS:

* 2,3,7,8-Tetrahylorodibenzo-1-dioxin (TCDD): 1 ppb Maximum

*Certification for TCDD content is to be furnished by the supplier for each batch of TCP.